

Receiving Tube Division
Owensboro, Kentucky

Quarterly Report No. 3

Interim Technical Report on

The Investigation of Synthetic and Substitute Materials in Domestic Supply for Use as Vacuum Tube Spacers.

For Period - 1 February, 1952 to 30 April, 1952

Date of Report - June, 1952

CONTRACT NO: Nober 52535

Submitted by:

J. C. Hickle Project Engineer

Approved by:

A. P. Hanse

Special Development Engineer

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College of Electrical Engineering	7
Cornell University	
Ithaca, Her York	

TECHNICAL PERSONNEL ADDED DURING THIRD INTERVAL

Richard J. Coswell

Date of Birth: February 20, 1926

Place of Birth: Detroit, Michigan

Education & Experiences BA (1947), MS (1949) Univ. of Southern California. General Electric Company, March 1949 Chemet Program 1949-1950

ABSTRACE

Evaluation of the physical characteristics of modifications of terrater and other asbestos based materials has continued.

Several new materials have been received and evaluated as to physical characteristics and performance in tubes.

New processing techniques for terratex are discussed and evaluated,

Results on tubes using many of the promising materials are presented and discussed with particular emphasis on case of degassing and level of cathode emission.

Technical man hours during this interval:

HM Broderick) RJ Coswell	155
Paul Doigan)	A
AP House	 27
JC Hickle	203
MA Chernin	322

The total expenditures on this contract through April 30, 1952 have been \$18,955.84.

PENE

The object of the work carried on under this contract is to find or develop a domestic material or materials to be used as vacuum tube spacers to replace or supplement the presently used mica.

The primary aim of the work will be to find a suitable material that will compete with mice in cost and case of fabrication.

The major problems in connection with the fulfilment of this centract are:

- Find a material of suitable strength and flexibility to allow standard assembly techniques to be used.
- 2. A material of such a mature that it may be fabricated to the standard tolerances used for mica at low cost and in large quantities.
- Physical and chemical properties such that it may be degased easily and not carry or contain elements or compounds detrimental to any of the other parts, partioularly the oxide-coated cathodes.
- 4. A material or materials which will give equivalent electrical characteristics without unjor design changes in the structure of the tubes e.g., (emission, transconductance, leakage, capacitance, shock resistance, wibration, microphonics and noise).

General Pactual Date

Our work during the third interval has continued to be devoted to evaluating the physical and chemical properties of asbestos-based and micabased sheet materials.

Variations in the asbestos-based material have been along two separate lines. First, the clay content of the asbestos sheet has been varied and the strength and other properties measured. Second, further attempts have been made to vary the processing of the regular terratex material in order to decrease the gas and moisture content. It is felt that any improvement of this type in the regular terratex could also be applied to the other asbestos-based materials. The investigation of the effect of different clay contents in the asbestos sheet was carried out on two different lots of material prepared as hand sheets by the T.A.P. Laboratory in Pittsfield, lisss. There was some indication that the physical properties varied somewhat with the percentage clay, and this is discussed in greater detail later in the report. Our investigations continued to explore the effects of various firing treatments followed by impregnating with various materials. These tests were performed on the sheet material or punched spacers and are discussed later in the report.

The evaluation of mice-based sheet materials was directed toward finding new types of material made available either commercially or experimentally and testing their physical properties and performance in vacuum tubes. These materials and the test results are discussed in the Detailed Section of this report.

Contacts were made with representatives of Johns-Manville, the supplier of the regular terrater rew material, in an effort to enligt their aid in obtaining machine-made samples with various clay contents. These contacts proved discouraging when they indicated that they would not undertake this type of work. This subject is being pursued with other paper manufacturers in an effort to obtain the desired materials.

Arrangements have been made to have gas analysis and thermal conductivity measurements made by the General Engineering Laboratory of the General Electric Company to determine what some of the limiting factors of the terratex and integrated mica materials might be. The results of these tests should be forthcoming in the next interval.

Detailed Factual Mata

The following descriptions identify the materials and variations investigated during the third interval of this contract.

```
Lot 137-1 - Terrater type material - 3.85 clay
              10
   137-3 -
                                - 2.35 clay
              .
                    .
 .
   137-4 -
                                 - 1.15 clay
* 137-5 -
                                 - 4.% olay
   137-6 -
             100
                                 - 6.35 "
     (All samples 0.011-0.013" in thickness)
Lot 138-2 - Terrater type material - 9.1% clay
n 138-3 -
                                 - 4.85
 138-4 -
                                 - 1.0% h
              -
                     " 138-5 -
              -
                                 - 2.05 R
     (All samples 0.020-0.022* in thickness)
Lot 141 - Type I.W. Quinterra (no clay) - 0.012" thick
Lot 142 - " " " - 0.015"
Lot 143 - Glass reinforced asbestos sheet (0.012-0.014")
Lot 1454- Terretex type material - 1.0% clay
         0.019-0.021" (Hand sheets)
Lot 1458- Terrater type material 2.0% clay
         0.018-0.020" (Head sheets)
Lot 2A - Regular machine-made terrater containing
          approximately IS olay
```

All of the above materials were treated with ethyl silicate using the solvent method.

Variations of processing of Lot 24 material were as follows: (All variations were preceded by a 5-simute baking at 5000.)

- (1) Baked 15 min at 400G followed by immersion in nitrocelluloge language solution.
- (2) Baked 15 min at 4000 followed by immersion in othylmilicate solution.
- (3) Heated in warmen to 5500 for 3 min., cooled 3 min, and repeated for total time of 24 min., followed by flooding under vacuum with nitrocallulose lacquer solution.
- (4) Same as (3) above, except flooded with ethyl-silicate solution.

Lot 121 - Silicone regin impregnated terrater (No ethyl-silicate treatment)

Integrated or Built-up Hice

Lot 1368 - GE Micenst treated with ethyl-silicate (0.006")

1328128 - Silicone remin treated integrated mice susplied by the Integrated Mice Corporation

PC2SH2B - Silicone regin treated integrated mice supplied by the Integrated Mice Corporation

NRIC Glass-bonded built-up mice flake supplied by the Mice Flate New England Mice Communy

The physical test results for these materials are tabulated in the following pages.

Degassing and Gas Content		Will investigate when	thicker meterial is grailable.		Analysis will be made.				Gas content seems to be similar to regular mics. May contain is purities that would injure costed cathodss.		
Funching Characteristics and General Reserves		Penches cleanly. Physical strength is very poor, but looks promising if greater thicknesses are possible.				Function olsenty. Function strength is good and contour is comparable to regular atom a		as Kellen, but is accentable.	As received material was too flacible and west. Resin did not seem to penetrate sheet, and resin carbonized on heat treatment. Other silloonse may be more suffable.	Material is very hard and stiff. Probably too hard to be punched economically in large quantities.	
	0.006	0.000	3	888 888 888			52.0	710		000 000 000 000 000	
P S	2	RR	র	88	6	S	ង	4		Bernts 12 18	
I.	As Rec'd	20 ath	*^3	K.A	As Reo'd	9	No. No. 1d	8	1	18 18 18 18 18 18 18 18 18 18 18 18 18 1	
Piring Jenn.	As Rec'd	7200	8	95	As Res'd	2006	N Pact	8	if in	48 Rec 40 60 60 60 60 60 60 60 60 60 60 60 60 60	
Interfal	Lot 1368	(0,006")			WZSRZB:	0.01)	CONT.	0.009)	Jot 121 Silvione Re- Impregnated Ferractor	Mica Plate	

Degassing and Gas Content				About the same gas content as previous samples of terrater.		No noticeable difference between these two materials and Lot 2A.
Punching Cherecteristics and General Remarks				Materials punch similarly to previous terratex samples. These sheets were thinger than we might expect to use in standard tube construction.		No appreciable difference in punching characteristics between the two materials. Seems to runch cleaner than the regular Lot 2A terratex.
	,		u u			,
Load at	0.18#	0.000	0.00	0.00 0.13 0.03 0.03	0.00 0.00 0.00 0.00 0.15	00.00 00
Bend	55 ₀	° Է ເຊ ເγ	48246	R1882	አ ድ ^ወ Էጸፈ	្ត ស្តង្គ «ឧក្សភ
Firing	As Received	หมาก สื	B60014	Received of National Section of National Secti	Received 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	As Received www.
Firing Temp.	_	\$\$\$\$\$ \$\$\$\$\$	A8 450 550 600	As 450 500 550 600	A8 450 600 600 A8 450 A8	550 600 500 500 600 600 600 600 600 600
Material	Lot 137-1 (0-011-0-013		Lot 137-3	Lot 137-4	lot 137-5	Lot 145-A (0.021- 0.023)

Degasaing and Gas Content	No noticeable difference between these two materials and lot 2A.		No great differences were observed as com- pared to Lot 2A.			ave as Pachings fusay n. ent
Punching Characteristics and General Remarks	No appreciable difference in punching characteristics between the two materials. Seems to punch classes than the regular lot 2A terrater.		These materials behaved similarly to previous lots of material in that the punchings were cleaner for the higher temperature firing. These samples had the best strength	any asbestos sheet teste is may be due to the fact e density was about 25 pe ester them lot 28.		This material does not seem to have asgood punching characteristics as the clay-containing materials. Punch have more of a tendency to have fusive outlines and poor hole definition. This may be partly due to different raw materials, or manufacturing processée of the sumplier
Load et	23×××	0000 8482	0000 34×2	8250 8250	34×3	00000000000000000000000000000000000000
Page 1	*******	% #%0	************	₹₽ ₩₩	255%	នកសសសងងង ដង្
Parise Time	As Received 400 5 min 500 5 5 500 5 5 600 5 5	2 As Received 400 5 500 5 600 5	Received	As Received 400 500 500 5	Received	៷ៜ៷ៜ៷ៜ៷ ៜ ៷ៜ
Piring	4 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8		4 6 8 8 8 8 8	\$ 55.00 \$ 50.00 \$ 50.00	3 8 8 8	ð - 3 - 6 - 3 - 6 - 6 - 6 - 6 - 6 - 6 - 6
Meterial	Lot 145-B (0.020- 0.021)	Lot 138-2	Lot 138-3	Lot 138-4	M /	Lot 141 (0,012")

. . .

d Degassing and Gas Content	the the the the out- This		Commentes	addition of th	seems to increase the strength and stiffness, but when the material is fired the second time at tem- peratures of 4000 or above, these offects are almost lost. No improve-	ment in gas content or ease of degassing was observed with the samples.	
Punching Cherecteristics and General Remarks	This material does not seem to have as good punching characteristics as the clay-containing materials. Funchings more of a tendency to have fuzzy outlines and poor hole definition. This may be cartly due to different revenues to ampliar.	Material was too thin and weak to be usable.	Bend Lond &t	22° 0.24 20 0.24 17 0.31 15 0.23	18 0.22 stiffy 18 0.24 is ful 17 0.22 peratu 17 0.23 effect	25 0.18 degr 25 0.19 sam 23 0.20	29 31 26 0.15 21 0.17
Bend Load at	«81448888888888888888888888888888888888		After Impregnation Town. Time	1750 15 Mtn	300 15	27 007 # # #	500
Firing Firing Temp. Time	4000 450 500 500 500 550 600 100 100 100 100 100 100 100 100 10	Not Tested	Before Limbremation Temp.	2000 5 mtn 550 550 550 550 550 550 550 550 550 55	000 000 000 000 000 000 000 000 000 00	2009 2009 2009 2009	2500 2500 2500 2500 2500 2500 2500 2500
Material	Lot 142 (0.015")	Lot 143	Materia	Lot 2A Terratex	Impregnated while hot with ethyl		

The preceding physical data indicate that the samples of M2SH2B and PC2SR2B integrated mica have physical properties suitable for vacuum tube spacers. Because the thickness of these materials is more uniform, they would be somewhat easier to handle in the normal manufacturing operations.

The data on the terrater materials with various percentages of clay seem to indicate that there is some variation of strength with clay content. However, these were hand sheets and do not necessarily indicate what we might experience on machine-made material. In general, the strength and stiffness increased with the percentage of clay. "

No apparent advantages are evident for the no-clay asbestos materials and they apnear to have poorer punching characteristics.

The effect of firing the terrates and giving it a second ethyl-silicate treatment seems to be desirable from the strength standpoint, but may be objectionable in tubes.

Tube Date

The JAN test limits for the two tube types for which data are presented are as follows:

-	RAKE
3320	2024

TO 124T

-Ig - 0,1 m 15 . 3-12 m

Se = 3500-6500 to 5.5 Se - 31.90 who -Ig = 2.25 tm 10 - 7-14 m

Sa 6 4500-6500 value 11 Sm = 4000 tubos

14 - 50 m

Type 30-61 (9654) tubes were made with the following spacer materials.
All camples were made from Lot 24 terretor.

Group 1 - Flain terrater

Group 2 - Alg03 sprayed terretex

Group 3 - Vegum fired nitrocallulose lacquer-impregnated terretex

Group 4 - Air behind nitrocellulose lacquer-treated terretex

Group 5 - Air baked sthyl-cilicate-treated terrater

Group 6 - Control group - regular wice

There was an average of 12 tubes in each lot, but data for only 5 of each lot are presented to show the general results for each group.

	9	ROUP I			9	BOUP 2			CRO	UP 3	
-Ig	Ip	S	5.5 Sm	-Ig	In .	S	5.5.8	-14	In	Sm	5.5.80
0.3	7.4 8.5 3.8 6.7 4.0	4850 5520 2380 4310 2330	3780 3530 1450 3040 1080	0 0 0.05	5.0 4.4 4.25 5.25 5.8	2560 2450 2470 2430 4060	1070 490 1060 1050 2320	0 0 0 0	4.4 6.65 4.5 6.8 5.0	2480 4050 2930 4130 2960	1360 2090 1590 3120
_	<u>0</u>	ROUP 4	• •		3	ROUP 5	Ť e			UP 6	
-Ig	Ip	Sm	5.5 Sm	-Ig	In	S	5.5 Sp	1	g In	<u>Sp</u> .	5.5 Sm

All of the tubes, including the control group, were unacceptable because of low transcenductance and reduced transcenductance. Group 5 was very close to the control group for this small test and might prove to be almost identical on a larger test. No conclusions have been drawn for the poor results on this group of tubes. Further tests will be necessary to determine the possible cause of these results.

Tests were made on type SD-6C (12AT7) tubes which were processed on the bench exhaust equipment unless otherwise noted.

The following results were obtained on type SD-600 using regular mice, 128128, and PCSSH2D integrated mice as spacer materials.

	REGI	ILAR I	LICA			1	28H2B	INTEUR	TED MICA		
-Ig	In	Sm	11 8	I I		عاد:	P	<u>8</u>	11 Sp	Is	
0	10.3	5890	5580	130		1.9	9.4	5330	4860	38	
0	8.3	5580	5080	108		0.9	9.0	5200	4600	54	
0		5240	4490	100		1.5*	9.3	5030	4060	38	
0.02		5890	5450	100			×9.6	5270	4750	54 38 85	
0		5640	5160	102		1.70		5160	4400	65	
					NET TOTAL ATTEST					7	

			118				
1.7* 2.4* 2.15* 2.00* 2.1*	9.7 9.5 9.3	5200 5170 5180	4700 4680 4700	97 65 55 90 88	*Indicates to gas.	lenkage	as opposed

The results of this test show the high leakage characteristics of the two integrated mica materials. Also, somewhat reduced emission is apparent, particularly with the M2SH2B material. This may be due to greater thermal conductivity of the material or to a nartial poisoning of the oxide-coated cathode. Fig. 1 shows the effect of heater voltage on the transconductance of these tubes

and verifies the results shown here. Further tests will be necessary to determine which of the above mentioned factors is causing these results.

In an attempt to minimize the interelement leakage, additional type SD-600 tubes were made with M2SH2B spacers that were given a special degrecing, air firing, and MgO spray.

r		REGULAR MICA							INTEGRATED MICA		
	-13	ID	Sm	11Sm	Is	9	-Ig	M2SH2B	Sm	11Sm	Is
	0	10.0	5010	4540	90	¥	0	9.3	4660	4300	60
	0	9.0	4900	4350	98		0	9.45	5120	4340	92
	0	8.4	4650	4360	80		0	8.2	4240	3590	29
	0	9,8	4830	4640	103	C	.02	7.9	4410	3430	25
	0	8.0	4460	4120	65		0	12.2	4960	4490	65

A complete analysis of 19 tubes in each lot (38 sections) gave the following median values:

	-Ig	<u>Ip</u>	Sm	11Sm	Is
Regular Mica M2SH2B	0		³⁴ 4900 4800		

It was encouraging to observe the marked reduction in interelement leakage in these tubes, but the reduced emission characteristics are still a cause for concern.

A third test was undertaken to determine the results of these materials in tubes produced on automatic exhaust equipment. The special processing of the integrated mica spacers was applied to this test.

RTGULAR MICA							2SII2B	INTEGR	ATED M	ICA
	-Ig	Ip	Sm	<u>118m</u>	Is	-Ig	In	Sm	115m	Is
		10.1	-		100	 0.35	10.0	5310	5010	77
-	0.02	10.25	5240	4790	102	0.30	11.2	5650	5250	62
	0	10.85	5180	4830	87	0.02	9.1	4860	4460	50
	0.01	10.8	5040	4860	90	0.08	8.7	4630	4100	50
	0	10.0	5230	5030	95	0.30	9.35	4150	7880	30

PC2	BH2B	NYDER	ATED M	<u>ICA</u>
-18	10	S	113	Is
0.30	11.2	5190	4840	65
0.02	10.1	5320	4990	72
0.10	9.9	5160	4950	75
0.05	10.5	5190	4950	80
0.12	10.45	5420	5120	80

A complete analysis of ambroximately 20 tubes in each lot (40 sections) gave the following median values:

æ			-Ig	In	Sm	115	Le
	Regular M2SH2B	Mica	0.05	10.0	5150 5000	4850	95 55
	PC2SH2B		0.10	10.2	5150	4850	75

Again the integrated mica materials resulted in somewhat lower emission fracteristics with the PC2SH2B material being superior to the M2SH2B. More damental information will have to be obtained before conclusions as to the use of these results can be drawn.

Tests on asbastos-based materials, i.e. terratex, were also conducted on e SD-60 (12AT7) tube type with the following results:

Group 1 - Regular mica, control group

2 - Plain terrater

3 - Vacuum fired, ethyl-silicate impregnated terratex

GROUP 1							GROUP 2			
-Ig	Ip	Sm	11Sm	Is	i	-Ig	Ip	Sm	118m	Is
0.01	9.9	5060	4720	90	a € }	0.04	10.5	5570	4340	105
0.05	9.7	4900	4360	80	P	₹0.02°	9.0	4980	4630	90
0	9.1	4620	4220	85		0.03	9.1	5110	4540	110
0	9.0	4820	4400	95		0.04	10.2	5150	4820	105
0.05	9.7	4970	4750	75		0.02	9.7	5300	4930	100

	- 1	GROUP	3	1	
-Ig	Ip	Sm	11Sm	<u>le</u>	
0	9.2	5040	4620	90	
0.03	10.0	5320	4860	100	
0.01	9.5	5000	4590	85	
0.02	9.3	5250	4730	105	
0	9.1	5130	4720	100	

Some improvements in gas content and ease of degassing were observed during the exhaust processing of the Group 3 tubes over the Group 2 tubes. The initial test data are encouraging, and life test information will be obtained.

In an effort to decrease the moisture content and keep it low, two silicone water-proffing agents were used to treat the terrater. The materials were "Dry Film" and "Soda T," both obtainable from the General Electric Company's Chemical Department.

No improvement in ease of degassing the tube was apparent during the exhaust processing. Tests on the tubes showed the Dry Film treated terratex-spaced tubes to have no emission. The Soda T treated terratex-spaced tubes compared with other terratex tubes and regular mica. No benefits were apparent from this added treatment.

Because Lot 138 terratex samples had been the best of the terratex materials with various percentages of clay added to the asbestos, type SD6CF (12AT7) tubes were made to determine what differences in degassing and tube characteristics might be obtained with these variations. These tubes were identified as follows:

Group 1 - Regular Mica, control Group
2 - Terratex with 9.1% Clay

3 - Terratex with 4.8% Clay

4 - Terratex with 1.0% Clay

5 - Terratex with 2.0% Clay

lie difference in case of degassing or gas content was discerned during the exhaust processing of the terratex spaced tubes. The following electrical test data were obtained:

					79					
	9	ROUP 1			1			CROUP 2		
-Ig	Ip	S	115m	Is		-Ig	Ip	Sm	1.1Sm	Is
0.03	9.6	4760	4340	80		0.25	9.8	4880	4440	75
0	9.6	4860	4620	65		0.02	8.1	4520	4020	65
0	9.7	4750	4500	80	9	0	8.4	4670	4350	35
0.25	9.1	4650	3070	80		Ŏ	7.6	4330	3880	50
0	8.1	4310	3800	65	•	o o	11.9	4310	3980	72
GROUP 3				GROUP 4						
-Ig	Ip	Sm	118	<u>Ie</u>	3	-Ig	, ID	Sm	11Sm	<u>Is</u>
0	8.75	4500	4130	58		0.10	8.3	4130	3600	30
0.05	9.2	4820	4310	62		0.12	8.4	4560	4020	45
0	8.1	4070	3730	50		0	75	4220	3830	49
· 0 ·	8.8	4570	4210	. 62		0.10		3910	3500	40
0	8.7	4760	4950	82		0	8.2	4540	4110	50
	:				GROUP	5			•	•
		1	-Ig	In	88	118	Is			
* × ×		. J	0	7.4	4400	3950	25		*	
,		100	0	8.7	4610	4320	70			
			ŏ	8.42	4640	4310	68			
			ŏ	8.2	4450	100				
						3800	45			
			. 0	9.2	4890	4480	80			

The results on this test were somewhat erratic, and since the number of tubes was small (less than 10 tubes/group), it is difficult to draw definite conclusions. The data for all the tubes does indicate that possibly Group 5 was the best of the terratex materials. More tests with larger numbers of tubes will be needed to establish this conclusively. These tests will be made when larger samples of sheet material are available.

Type SD-6CC (12AT7) tubes were also made to commere the NEMC plate with regular sheet mica. The data are not presented because the tubes with the NEMC material spacers had practically no emission. Further investigation of this material does not seem warranted at this time.

From the preceding data, several materials show promise, and further testing of these will continue. The initial test results on the M2SH2B and PC2SH2B materials indicate the need for more tests. Life tests on tubes using these materials should be made. Also, life test data will be taken on tubes using the vacuum fired, ethyl-silicate impregnated terratex since the tubes look satisfactory initially. Further tests on the variation of clay content in the terratex type material will continue in an effort to obtain the best overall characteristics.

Conclusions

The two types of integrated mice, M28M2B and PC28M2B, gave tubes that were satisfactory initially but not as good as the regular mice control tubes. The need for for thorough degreesing and suraying the surface to reduce leakage on those two materials is evident. The PC28M2B material genears to give results more nearly like short mice than does the M28M2B.

Additional tests on the SD-60 (12AT7) and other types, particularly life tests, are necessary to determine limitations of the integrated mica materials.

Results on the NFMC Mica Plate were discouraging, and no advantages over the two integrated mica materials are evident. No additional tests seen warranted at this time.

Some improvement in ease of degassing and gas content has been made on the terratex material by a process of vacuum firing and a second ethyl-silicate imprognation. Further improvement is still desirable.

Variations in clay content of the terratex sheets affect the physical properties of this material and additional samples should be obtained.

Work to be Performed During Next Interval

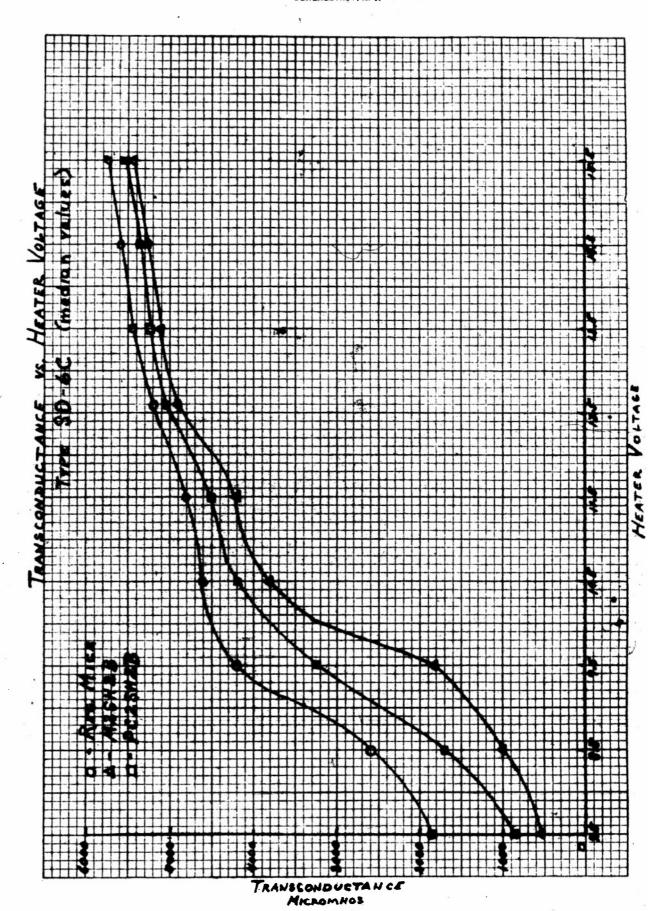
Additional evaluation of the two integrated mica materials with particular emphasis on the thermal conductivity and gas analysis measurements.

Continuation of the investigation of new processing of the .020" terrater in an effort to further increase physical strength and ease of degassing.

Analyzation of the gases given off by the terratex under high vacua at elevated temperatures to learn what gases are present, thus indicating what constituents might be eliminated.

Evaluation of machine made samples of terratex with varying clay content 'for effects on smacer material.

Continuation of investigation of new materials that indicate promise as tube sources.



F19. 1